THE EFFECT OF SOLVENT EXTRACTION ON THE REFLECTANCE OF COAL AND COAL-OIL MIXTURES

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INTRODUCTION

The commercial classification of coal is often based upon its rank. Technological properties, such as the potential for liquefaction, also vary with the rank of the coal since this is a measure of the average chemical composition. Coal rank can be determined from the reflectance of the coal using a microscopic method.

In many coal liquefaction processes the coal is co-processed with heavy oil or pitch. The solid residues from the process can contain unreacted coal, partially reacted coal intermediate products and semicoke. The semicoke could have been formed from the coal, the oil or pitch or from the liquid products of the process. A detailed petrographic analysis of these solid products can provide useful information about their nature and source.

Petrographic analysis is done by mounting crushed coal or residues in epoxy resin, polishing the surface and examining it microscopically using reflected light and oil immersion objectives. Any oil or pitch present in the sample will dissolve in the oil used to coat the objective lens and both obscure the image and more importantly, prevent accurate measurement of the reflectance of the sample. The oil is usually removed by Soxhlet extraction; pentane, tetrahydrofuran (THF) and toluene are the most common solvents.

One way to determine whether coal found in the solid residues is unreacted, or partially reacted is by measuring its reflectance and comparing this with the reflectance of the coal feedstock components from which they are derived. Changes in reflectance are related to changes in the composition (rank) of the coal. A major problem (1) with this approach is that changes in reflectance may have occurred while the coal was stored as a slurry with the oil and/or during the solvent extraction.

The solvent extraction process does not appear to affect the reflectance of bituminous coals. Some previous observations seem to indicate that the reflectance of the coal was affected by the oil or by the solvent extraction, but this was not conclusive. A series of experiments was done to directly determine the effect of soaking in oil and subsequent solvent extraction on the reflectance of low rank coals (4).

RESULTS AND DISCUSSION

Two lignites from southern Saskatchewan were used, from the Boundary Dam Mine (BDM-8) and the Coronach Mine (CM-8). They were of rank lignite B, and were chosen as they contained relatively large amounts of inertinite and exinite (Table 1) so that measurements of reflectance on all three major maceral groups could be made. The coals were ground to less than 20 mesh and separated, by riffling, into seven samples. Three samples were mixed with heavy oil (Lloydminster) and stored for 24 days at room temperature and 30°C for 7 days. They were extracted with a solvent (Soxhlet method) until the filtrate, after the coal had stood overnight, with stirring, in solvent, was colourless. Three solvents were used, pentane (168 hours extraction time), toluene (76 hours) and tetrahydrofuran (52 hours). Three coal samples that had not been treated with oil were solvent extracted with the same solvents for the same time. One coal sample was left untreated.

The loss in mass of the coals during the extraction process was determined for each sample. It was about 20%, which is high (3), but since some of this loss was due to handling there is no quantitative significance to the differences in the loss of mass.

TABLE 1

PETROGRAPHIC ANALYSES (%)

	BDM-8	CM-8
Huminite	72	56
Exinite	9	4
Inertinite	25	21
Mineral matter	10	3

Reflectances

The coals were set in epoxy resin and polished. Reflectances, at 546 nm, were measured against optical glass standards using a Leitz Orthoplan-pol, MPV compact microscope/photometer system. A total of 100 random measurements were made for each type of huminite and 75 for the other, rarer, macerals. The mean random reflectance, rather than the maximum reflectance, is usually determined for low rank coals because huminites and liptinites are isotropic.

The reflectance of huminite is usually measured since it is the most common, and the most uniform, maceral. The results for dark huminite (eu-ulminite A) in the CM-8 coal are given in Table 2. The reflectances are quoted to 3 figures, but are statistically valid to two figures only. The reflectance of CM-8 has not been affected by either extraction with a solvent or by soaking in oil followed by extraction.

TABLE 2

MEAN RANDOM REFLECTANCES, DARK HUMINITE

	BDM-8	CM-8
Feedstock	0.253	0.246
Pentane	0.262	0.252
Oil/pentane	0.274	0.254
Toluene	0.292	0.263
Oil/toluene	0.286	0.265
THF	0.289	0.246
Oil/THF	0.285	0.247

There does seem to be a pattern of increasing reflectance with severity of extraction (temperature of extract on and solvating power of solvent) with the BDM-8 coal, but the variations are within the average standard deviation of 0.03. There is some evidence from the reflectance data that the dark huminite in BDM-8 feedstock consisted of two subgroups, with mean random reflectances of 0.18 and 0.27. The amount of the lower reflectance subgroup decreased after solvent extraction, but again these differences, while consistent, are well within the statistical error.

Measurements were also made on light huminite, exinite and inertinite. Solvent extraction or soaking in oil had no discernable effect on the mean random reflectance of any of these macerals. There is a general relationship between reflectance and aromaticity (5). Solvent extraction with pentane, toluene or THF, while it removed some coal material (up to 20%) from the low rank coals had no effect on the overall degree of aromaticity of the coal.

CONCLUSIONS

The solid coal residues from coal liquefaction processes often show a change in reflectance. This change is, with low rank coals, not due to soaking in oil or sub-

sequent solvent extraction to remove the oil. It is a measure of the chemical changes in these coal particles during the liquefaction process and can be used as a measure of the severity of the reaction that these particles have undergone.

LITERATURE CITED

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